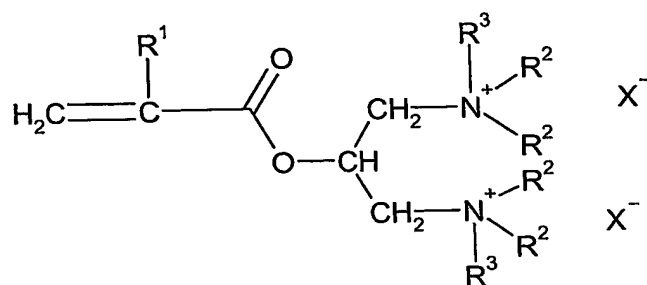


Claims

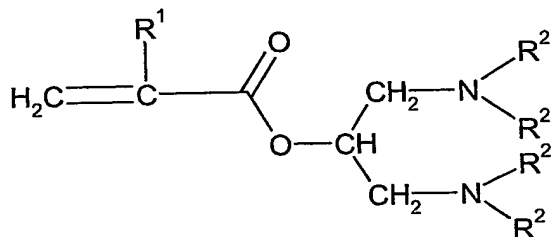
1. Process for the manufacture of a (meth)acrylate di-ammonium salt of formula (I)



(I)

wherein  $R^1$  represents hydrogen or methyl, each  $R^2$ , independently, represents an alkyl comprising from 1 to 4 carbon atoms, each  $R^3$ , independently, represents an alkyl or an aralkyl and each  $X^-$ , independently, represents an anion, comprising

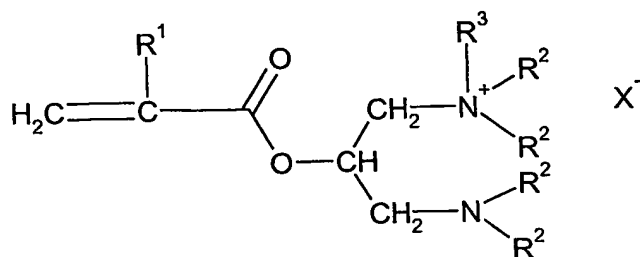
- (1) the reaction of the di-amino (meth)acrylate of formula (II)



(II)

with at least one alkyl or aralkyl derivative of formula  $R^3X$  in an organic solvent containing at most 5000 ppm, preferably at most 1000 ppm, of water and wherein the compound of formula (I) has a solubility at 25 °C of less than 1 g/100 g of solvent, preferably of less than 0.5 g/100 g of solvent, and wherein the solubility of the corresponding amino-(meth)acrylate ammonium salt of formula (V)

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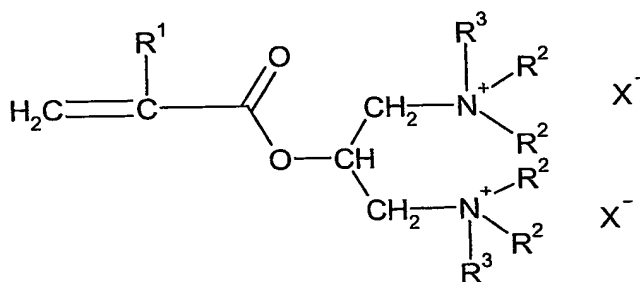


(V)

has a solubility at 25 °C of at least 20 g/100 g of solvent ; and

(2) the separation of the compound of formula (I) from the reaction mixture without dissolving it in water, the compound of formula (I) being separated from the reaction mixture in the form of a solid product comprising, per mole of the compound of formula (I), less than 0.1 mole, preferably less than 0.05 mole and more preferably less than 0.01 mole of the compound of formula (V).

- 10 2. Process for the manufacture of a polymer comprising units derived from at least one (meth)acrylate di-ammonium salt of formula (I)

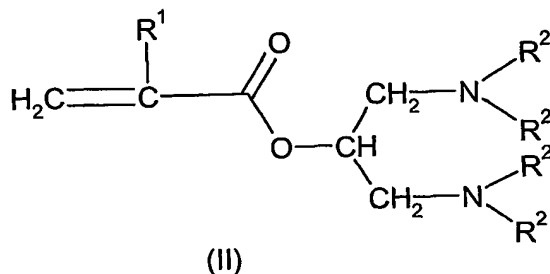


(I)

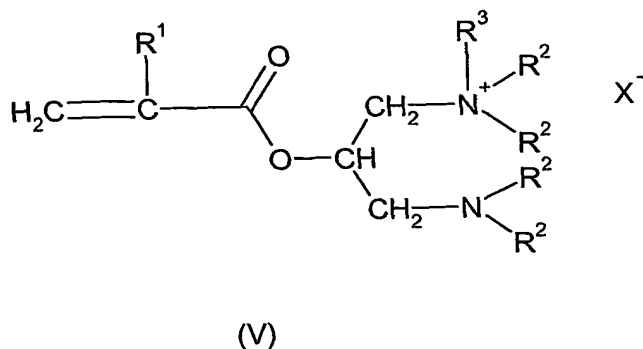
wherein  $R^1$  represents hydrogen or methyl, each  $R^2$ , independently, represents an alkyl comprising from 1 to 4 carbon atoms, each  $R^3$ , independently, represents an alkyl or an aralkyl and each  $X^-$ , independently, represents an anion, comprising

- (1) the reaction of the di-amino (meth)acrylate of formula (II)

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with at least one alkyl or aralkyl derivative of formula  $R^3X$  in an organic solvent containing at most 5000 ppm, preferably at most 1000 ppm, of water and wherein the compound of formula (I) has a solubility at 25 °C of less than 1 g/100 g of solvent, preferably of less than 0.5 g/100 g of solvent, and wherein the solubility of the corresponding amino-(meth)acrylate ammonium salt of formula (V)



has a solubility at 25 °C of at least 20 g/100 g of solvent ;

(2) the separation of the compound of formula (I) from the reaction mixture without dissolving it in water ; the compound of formula (I) being separated from the reaction mixture in the form of a solid product comprising, per mole of the compound of formula (I), less than 0.1 mole, preferably less than 0.05 mole and more preferably less than 0.01 mole of the compound of formula (V), and

(3) the polymerisation of at least the compound of formula (I) contained in said solid product to achieve said polymer.

3. Process according to claim 2, wherein step (3) comprises the co-polymerisation of at least 12 % by weight, preferably of at least 20 % by weight, and more preferably of at least 25 % by weight of the compound of formula (I) contained in said solid product with at the most 88 % by weight, preferably at the most

80 % by weight, and more preferably at the most 75 % by weight of at least one further monomer, the compound of formula (I) being co-polymerised preferably in an amount of less than 99 % by weight, more preferably in an amount of less than 85 % by weight with said further monomer.

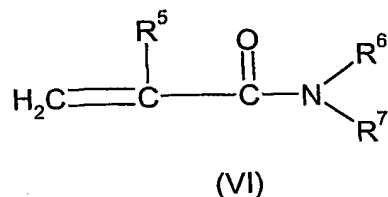
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4. Process according to claim 2 or 3, characterised in that said process is a radical co-polymerisation of a water-based solution of monomers polymerised by emulsion or suspension techniques.

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5. Process according to any one of the claims 2 to 4, wherein step (3) comprises the co-polymerisation of from 1 to 99 parts by moles of the compound of formula (I) contained in said solid product with from 1 to 99 parts by moles of at least one acrylamide monomer of formula (VI)

15



wherein  $\text{R}^5$  is hydrogen or methyl,  $\text{R}^6$  and  $\text{R}^7$  are, independently, hydrogen, alkyl comprising from 1 to 6 carbon atoms, optionally substituted by one or more hydroxy or alkoxy groups.

20

6. Process according to any one of the claims 1 to 5, wherein the solvent is an aprotic dipolar solvent, in particular a solvent selected from the group consisting of acetone, methylethylketone, ethylacetate, nitromethane, acetonitrile or mixtures thereof, the solvent being preferably acetonitrile.

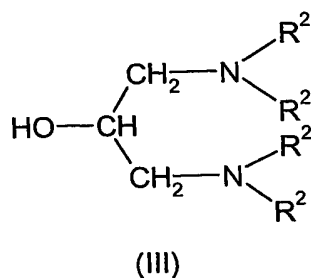
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7. Process according to any one of the claims 1 to 6, wherein the separation of the (meth)acrylate di-ammonium salt of formula (I) from the reaction mixture is done by mechanical separation, in particular by filtration or centrifugation.

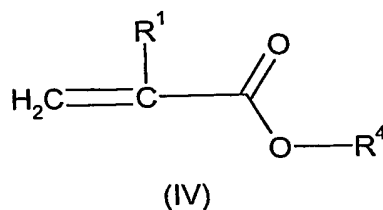
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8. Process according to any one of the claims 1 to 7, wherein the reaction mixture obtained after step (2) is recycled.

9. Process according to any of claims 1 to 8, wherein the di-amino (meth)acrylate of formula (II) used in step (1) is prepared by the transesterification of a 1,3-di-amino-2- propanol of formula (III)



- 5 wherein each  $R^2$ , independently represents an alkyl comprising from 1 to 4 carbon atoms, with a (meth)acrylate of formula (IV)



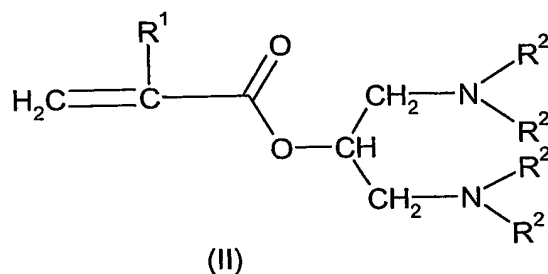
wherein  $R^1$  represents hydrogen or methyl and  $R^4$  represents an alkyl comprising from 1 to 4 carbon atoms, in the presence of a lithium-catalyst.

10

10. Process according to any one of the preceding claims, wherein X is Cl.
11. Process according to any one of the preceding claims, wherein the di-amino (meth)acrylate of formula (II) is allowed to react in step (1) with more than 2, preferably with more than 2.1, equivalents of said alkyl or aralkyl derivative of formula  $R^3X$ .
12. Process according to any one of the preceding claims, wherein the reaction of step (1) is effectuated in an amount of between 500 and 5000 g of solvent per mole of the di-amino (meth)acrylate of formula (II).
13. Solid product obtainable by the process according to claim 1, or by the process according to any one of the claims 6 to 12 when dependent on claim 1, containing the (meth)acrylate di-ammonium salt of formula (I) and, per mole of

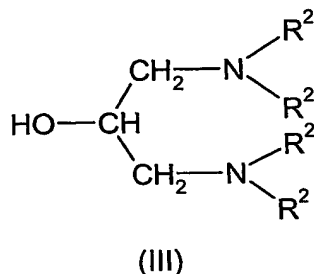
this salt, less than 0.1 mole, preferably less than 0.05 mole and more preferably less than 0.01 mole, of the corresponding amino-(meth)acrylate ammonium salt of formula (V).

- 5 14. Polymer obtainable by the process according to any one of the claims 2 to 5, or by the process according to any one of the claims 6 to 12 when dependent on any one of the claims 2 to 4, containing units derived from at least one (meth)acryl di-ammonium salt of formula (I) and, per n units derived from this di-ammonium salt, less than  $0.1 \cdot n$ , preferably less than  $0.05 \cdot n$  and more  
10 preferably less than  $0.01 \cdot n$  units derived from at least one amino-(meth)acrylate ammonium salt of formula (V).
- 15 15. Polymer obtainable by the process according to claim 3, or by the process according to any one of the claims 4 to 12 when dependent on claim 3, containing at least 12 % by weight, preferably at least 20 % by weight, and more preferably at least 25 % by weight of units derived from at least one (meth)acryl di-ammonium salt of formula (I) and at the most 88 % by weight, preferably at the most 80 % by weight, and more preferably at the most 75 %  
20 by weight of units derived from at least one further monomer, the polymer containing preferably less than 99 % by weight, more preferably less than 85 % by weight of at least one (meth)acryl di-ammonium salt of formula (I).
16. Use of a polymer according to claim 14 or 15 as flocculant.
- 25 17. Process for the manufacture of a di-amino (meth)acrylate of formula (II)

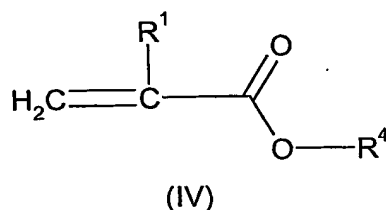


by transesterification of a 1,3-di-amino-2- propanol of formula (III)

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wherein each  $R^2$ , independently represents an alkyl comprising from 1 to 4 carbon atoms, with a (meth)acrylate of formula (IV)



- 5            wherein  $R^1$  represents hydrogen or methyl and  $R^4$  represents an alkyl  
              comprising from 1 to 4 carbon atoms, in the presence of a lithium-catalyst.
18.    Process according to claim 9 or 17, wherein the lithium catalyst is chosen from  
           $Li_2O$ ,  $LiOCH_3$ ,  $LiOH$  and their mixtures.
- 10
19.    Process according to claim 9, 17 or 18, wherein the transesterification is done  
          at a temperature not exceeding 120 °C, preferably at a temperature not  
          exceeding 110 °C, the transesterification being preferably done at a  
          temperature of at least 80 °C.
- 15
20.    Process according to any one of the claims 1 to 12 or according to any one of  
          the claims 17 to 19, wherein  $R^1$  is methyl.
21.    Process according to any one of the claims 1 to 12 or according to any one of  
          the claims 17 to 20, wherein  $R^2$  is methyl.
- 20

22. Process according to any one of the claims 1 to 12 or according to any one of the claims 17 to 21, wherein  $R^3$  is methyl.
23. Process according to any one of the claims 1 to 12 or according to any one of the claims 17 to 21, wherein  $R^3$  is benzyl.
24. Process according to any one of the claims 9 to 12 or according to any one of the claims 17 to 23, wherein  $R^4$  is methyl.